SPECIFICATION

FINE PARTICULATE SILVER POWDER AND PRODUCTION METHOD THEREOF

Technical Field

The invention of this application relates to a fine particulate silver powder and a production method of the fine particulate silver powder.

Background Art

Conventionally, for the production of a silver powder, wet reduction process has been adopted in which an aqueous solution of silver ammine complex is produced with a silver nitrate solution and ammonia water, and an organic reducing agent is added to this as described in Patent Document 1. In late years, these silver powders are used for forming electrodes and circuits of chip parts, plasma display panel, etc. as main field of application.

[Patent document 1]: Japanese Patent Laid-Open Publication No. 2001-107101

Therefore, as for such electrodes and circuits, drastic fine production of the electrodes and circuits are demanded, and high reliability has come to be demanded along with high density and high accuracy of electric wiring.

Disclosure of the Invention

Problems to be solved by the invention

However, the powder particles of silver powder obtained by this conventional production method have an average particle diameter of primary particles D_{IA} usually exceeding 0.6 μm and an average particle diameter D_{50} by laser diffraction scattering particle size distribution measurement method exceeding 1.0 μm, and the aggregation degree expressed by D_{50}/D_{IA} exceeds 1.7 in actual condition. Therefore they were unsuitable for the recent fine-pitched circuit formation, and made a significant factor of decreasing product yield.

In the meantime, the following problems have arisen from a viewpoint of usage of silver powders. It has been conventionally considered that a low crystalline silver powder is desirable for achieving a high degree of sinterbility at a low temperature because non-sintering or low-temperature-sintering type in which the heat temperature is 300°C or less has been largely used in the circuit formation using a silver paste. However, fast reduction reaction systems have had to be adopted in the production condition to obtain a low crystalline silver powder, and, as a result, only silver powders which have low crystallinity but are remarkably aggregating have been able to be obtained.

Under these circumstances, silver powders which are unprecedentedly fine particulate silver powders and have a dispersibility more approximate to monodispersibility resulting less aggregation and also have excellent low-temperature sintering properties have been desired to be provided in the market.

Accordingly, the present inventors have conducted intensive studies and made the best use of inventiveness in the production method based on a conventional production method in which an aqueous solution of silver ammine complex is obtained by mixing and reacting an aqueous silver nitrate solution and ammonia water, and an organic reducing agent is added to this to allow reduction and deposition of silver particles which are then filtered, washed and dried. As a result, it has been enabled to obtain fine particulate silver powders of a level unattainable by a conventional production method, and further, a production method for obtaining the fine particulate silver powders stably at a high yield. Hereinbelow, the present invention will be described as divided into the "fine particulate silver powder" and the "production method".

<Fine particulate silver powder>

First, the fine particulate silver powder according to the present invention is described. The fine particulate silver powder according to the present invention is mainly characterized in that it has the following powder properties a. to c. As for these powder properties, they are enumerated as the most conspicuous properties and common in the fine particulate silver powder according to the present invention under current powder measurement techniques. Hereinbelow, each of the properties is described.

The property a. is that the average particle diameter D_{IA} of the primary particles obtained by image analysis of

a scanning electron microscope image is 0.6 μm or less. Here, the "average particle diameter D_{IA} of the primary particles obtained by image analysis of a scanning electron microscope image " is an average particle diameter obtained by image analysis of an image of the silver powder observed by a scanning electron microscope (SEM) (wherein it is preferable to observe at a magnification of 10,000 times in the case of a fine particulate silver powder according to the present invention and at a magnification of 3000-5000 times in the case of a conventional silver powder.). The image analysis of the silver powder observed by a scanning electron microscope (SEM) in the present specification is performed by obtaining average particle diameter D_{IA} by using IP-1000PC manufactured by Asahi Engineering Co., Ltd. and conducting a round particle analysis assuming the circular threshold as 10, and the overlapping degree as 20. Because the average particle diameter D_{IA} obtained by the image analysis of the observed image is directly obtained by the SEM observation image, the average particle diameter of the primary particles can be surely obtained. The D_{IA} as used in the present invention is mostly falls within the range of 0.01 μm to 0.6 μm, as far as the present inventors observe it, but more minute particle size may be observed in reality, and therefore, no specific lower limit is described on purpose.

As for the property b., because the fine particulate silver powder according to the present invention shows a high dispersibility never shown by the conventional silver powders, "aggregation degree" as an index of showing this

dispersibility is used. The "aggregation degree" as used in this specification is the value represented by D_{50}/D_{IA} using the above-mentioned average particle diameter D_{IA} of primary particles and the average particle diameter D_{50} by laser diffraction scattering particle size distribution measurement method. Here, D_{50} is particle size at 50% weight accumulation obtained by laser diffraction scattering particle size distribution measurement method, and the value of this average particle diameter D_{50} is not by directly and truly observing the diameter of the powder particles one by one but it can be said that an average particle diameter by assuming an aggregated powder particle as one particle (aggregated particle) is calculated. This is because powder particles of a real silver powder are usually considered not to be so-called monodisperse powder in which each individual particle is completely separated but in a condition in which several powder particles are aggregated. However, it is usual that the value of average particle diameter D_{50} becomes smaller as less aggregation of powder particles is present, and they are more approximate to monodisperse. D_{50} of the fine particulate silver powder to be used in the present invention is a range of about 0.25 μm to 0.80 μm, and a fine particulate silver powder is provided having an average particle diameter D_{50} of the range which has not been obtained at all by a conventional production method. The laser diffraction scattering particle size distribution measurement method as used in this specification is performed by mixing 0.1 g of fine particulate silver powder with ion-exchange water, and

dispersing it with an ultrasonic homogenizer (a product of Nippon Seiki Seisaku-sho Co., Ltd., US-300T) for five minutes and measuring with a laser diffraction scattering particle size distribution measuring apparatus MicroTrac HRA 9320-X 100 type (a product of Leeds & Northrup company).

In contrast, the "average particle diameter D_{IA} of the primary particles obtained by image analysis of a scanning electron micrograph" is an average particle diameter obtained by image analysis of an image of the silver powder observed by a scanning electron microscope (SEM) and the average particle diameter of the primary particles is surely observed without considering state of aggregation.

As a result, the present inventors have decided to take the value calculated by D_{50}/D_{IA} calculated from the average particle diameter D_{50} by laser diffraction scattering particle size distribution measurement method and the average particle diameter D_{IA} obtained by image analysis as the aggregation degree. That is to say, assuming that the D_{50} and D_{IA} values can be measured with same precision in a fine particulate silver powder of the same lot, the value of D_{50} allowing the state of aggregation to be reflected in the measured value is supposed to be larger than that of D_{IA} based on the theory stated above. The value of D_{50} approaches the value of D_{IA} limitlessly, and the value of D_{50}/D_{IA} which is the aggregation degree will approach 1 as the state of aggregation of the powder particles of the fine particulate silver powder is reduced. At the stage where the aggregation degree becomes 1, the powder can be said to be monodisperse powder in which

the state of aggregation of the powder particles is not present at all.

Accordingly, the present inventors have tried to examine the correlation among the aggregation degree, viscosity of fine particulate silver powder pastes produced with a fine particulate silver powder of different aggregation degree and surface smoothness of the conductor obtained by sintering. As a result, it has been found that an extremely good correlation can be obtained. As is understood from this, it can be judged that free control of viscosity of a fine particulate silver powder paste is possible by controlling the aggregation degree of the fine particulate silver powder used therein. Besides, it has been found that if the aggregation degree is maintained to be 1.5 or less, fluctuation of the viscosity of fine particulate silver powder paste, and the surface smoothness after sintering processing can be retained in an extremely small region. In addition, as the aggregation state is eliminated, the film apparent density of the conductor obtained by sintering the fine particulate silver oxide powder improves and, as a result, it comes to be possible to reduce the electric resistance of the formed sintered conductor.

In addition, when the aggregation degree is actually calculated, there are some cases where the value less than 1 is shown. It is considered that this is due to the assumption that D_{IA} used for calculation of aggregation degree is a truth sphere, and although the value of aggregation degree cannot be a value under 1 theoretically, it is supposed that such

a value of aggregation degree less than 1 can be obtained in reality because it is not a truth sphere.

The property c. is that crystallite diameter is 10 nm or less and there is a very close relationship between this crystallite diameter and sintering starting temperature. That is to say, in the comparison between the silver powders having equal average particle diameter, the sintering temperature can be lower as the crystallite diameter is smaller. Therefore, the fine particulate silver powder of the present invention having a large surface energy due to its small particle size, and having a small crystallite diameter of 10 mm or less can reduce the sintering starting temperature. Here, no lower limit is defined for the crystallite diameter, and the reason therefor is that a certain measurement error occurs depending on measuring apparatus, measurement condition and so on. It is difficult to demand high reliability in the measured values in the range where the crystallite diameter is less than 10 nm and if the lower limit is dared to be determined, it is supposed to be around 2 nm obtained as a result of study of the present inventors.

The fine particulate silver powders according to the present invention have the powder properties a. to c. as stated above, and from a viewpoint of sintering starting temperature, the fine particulate silver powder according to the present invention can be taken as a fine particulate silver powder having sintering properties lower than 240°C. No lower limit is particularly defined for this sintering starting temperature either, but it is almost impossible to attain

a sintering starting temperature less than 170°C and this is supposed to be said temperature corresponding to the lower limit in consideration of the study performed by the present inventors and general technique common sense.

Furthermore, tap bulk density of the fine particulate silver powder according to the present invention is as high as 4.0 g/cm^3 as an effect resulted by the powder properties mentioned above. The tap bulk density as used herein is measured by a method comprising accurately weighing 200 g of fine particulate silver powder, placing the powder in a measuring cylinder of 150 cm^3, tapping by repeating dropping at a stroke of 40 mm 1,000 times, and then measuring the volume of the fine particulate silver powder. This tap bulk density will be obtained as a higher value as the powder has theoretically more minute particle size, and it is in a state of higher dispersibility without aggregation among the particles. Considering that the tap bulk density of the conventional silver powders was less than 4.0 g/cm^3, this supports that the fine particulate silver powder according to the present invention is very fine and excellent in dispersibility.

<Production method of fine particulate silver powder>

The production method according to the present is a method in which an aqueous solution of silver ammine complex is obtained by contacting and reacting an aqueous silver nitrate solution and ammonia water, and an organic reducing agent is added to this to allow reduction and deposition of silver particles which are then filtered, washed and dried,

significantly characterized in that the reducing agent, silver nitrate and ammonia water are used in amounts so that they may be diluted after they are added. Conventionally, it was common to mix a reducing agent solution and a silver ammine complex aqueous solution in a tank at once and therefore, large amounts of silver nitrate, reducing agent and ammonia water should be added in order to make the silver concentration to be 10 g/1 or more, and otherwise productivity for the scale of facilities was not able to be secured.

The most important characteristic of the production method according to the present invention is that the concentration of the organic reducing agent after contacting and reacting a silver ammine complex aqueous solution and an organic reducing agent is low, and it is possible to decrease the organic reducing material which may be adsorbed and left on the surface of the powder particles of the generated silver powder, or taken in the powder particles in the growing process of the powder particles. Therefore, it is the most preferable to maintain the concentration of the organic reducing agent to 1 g/1 to 3 g/1 whereas the silver concentration is adjusted to 1 g/1 to 6 g/1 in the mixed solution.

Here, there is a proportional relationship between the silver concentration and naturally quantitatively a larger amount of silver powders can be obtained as the silver concentration is higher. However, when the silver concentration exceeds 6 g/1, there arises a tendency that the deposited silver particle becomes coarse particles, and the particle diameter will not be different at all from that

of the conventional silver powders and silver powders having high dispersion properties by the present invention cannot be obtained. In contrast, if the silver concentration here is less than 1 g/l, extremely fine particulate silver powder can be obtained but because the powder is too fine, oil absorption increases and the viscosity of the paste rises, which brings about necessity to increase the amount of the organic vehicle, and leads to decrease in the film density of the finally formed sintered conductor, and tendency of increase in the electric resistance. In addition, it will not satisfy the necessary industrial productivity.

And it is the most suitable condition for obtaining fine particulate silver powder according to the present invention in a high yield to maintain the concentration of the organic reducing agent to 1 g/l to 3 g/l while the silver concentration to 1 g/l to 6g/l. Here, the reason for adjusting the concentration of the organic reducing agent to 1 g/l to 3 g/l is to select it as the range most suitable for obtaining a silver powder of fine particles in a relationship with the silver concentration in the silver ammine complex aqueous solution. When the concentration of the organic reducing agent exceeds 3 g/l, the amount of the reducing agent liquid to be added to the silver ammine complex aqueous solution decreases, but the progress of aggregation of the powder particles of the reduced and deposited silver powder comes to be significant, and the amount of impurity contained in the powder particles (In this specification, the amount of impurity is taken as carbon content.) begins to increase

rapidly. On the other hand, when the concentration of the organic reducing agent is adjusted to less than 1 g/l, the total liquid volume of the reducing agent to be used increases and the amount of waste water treatment grows significantly, and it cannot satisfy the industrial economic efficiency.

The "organic reducing agent" as used herein is hydroquinone, ascorbic acid, glucose, etc. Among these, it is preferable to use hydroquinone for an organic reducing agent selectively. Hydroquinone is comparatively excellent in reactivity in the present invention in comparison with other organic reducing agents, and it may be said that it is the agent having a reaction rate most suitable for obtaining low crystalline silver powder having a small crystallite diameter.

And the other additives can be used in combination with the above-mentioned organic reducing agents. The additives as used herein are glue such as gelatine, amine-based polymeric agent, cellulose and so on and desirably they stabilize the reduction deposition process of silver powder and have functions to be a certain dispersing agent at the same time and can be used suitably selectively in accordance with the organic reducing agent, type of production process and so on.

And according to the present invention, it is desirable to adopt a method of contacting and reacting the resulting silver ammine complex aqueous solution and an organic reducing agent to reduce and deposit a fine particulate silver powder, wherein the silver ammine complex aqueous solution S_1 flows

in a certain flow path (referred to as "the first flow path" in the above and the following) and the second flow path b is provided which joins the first flow path a on its way as shown in Figure 1 and the organic reducing agent and an additive S_2, if required, are allowed to flow into the first flow path a though the second first flow path b and are contacted and mixed at the joining point m of the first flow path a and the second first flow path b to reduce and deposit a silver powder (hereinafter referred to as "interflow mixing method").

By adopting such interflow mixing method, mixing of the two liquids is achieved in the shortest time and the reaction proceeds in a uniform state within the system a, and therefore, powder particles of the uniform shape can be formed. In addition, because the amount of the organic reducing agent after mixing is small when observed as the whole solution, the amount of the organic reducing agent adsorbed and left on the surface of the powder particles of the reduced and deposited silver powder decreases. As a result, the amount of impurities adsorbed on each of the fine particulate silver powder obtained by filtering and drying can be reduced. This reduction of the amount of impurities adsorbed on the fine particulate silver powder also enables to reduce the electric resistance of the sintered conductor which is formed through a silver paste.

Furthermore, it is desirable to obtain a silver ammine complex aqueous solution at a silver concentration of 2 g/l to 12 g/l using a silver nitrate aqueous solution of 2.6 g/l

to 48 g/l when contacting and reacting a silver nitrate aqueous solution and ammonia water to obtain a silver ammine complex aqueous solution. The prescription of the concentration of a silver nitrate aqueous solution here is the same as prescribing the liquid volume of the silver nitrate aqueous solution and in consideration that the silver concentration of the silver ammine complex aqueous solution is adjusted to 2 g/l to 12g/l, the concentration and the liquid volume of ammonia water to be added thereto will be necessarily decided. Although clear technical reasons have not been clear in the present stage, a fine particulate silver powder showing the best production stability and stable in quality can be obtained by using a silver nitrate aqueous solution of 2.6 g/l to 48 g/l.

The fine particulate silver powder according to the present invention is fine as never present conventionally and has a high dispersibility and it can be understood that such a silver powder is never present in the conventional silver powders. In addition, it is enabled to efficiently obtain the fine particulate silver powder according to the present invention by adopting the production method described above.

Brief Description of the Drawings

Figure 1 is a drawing expressing the concept of mixing a silver ammine complex aqueous solution and a reducing agent;

Figure 2 is a scanning electron micrograph of a fine particulate silver powder according to the present invention;

Figure 3 is a scanning electron micrograph of a fine particulate silver powder according to the present invention;

Figure 4 is a scanning electron micrograph of a fine particulate silver powder according to a conventional production method; and

Figure 5 is a scanning electron micrograph of a fine particulate silver powder according to a conventional production method.

Best Mode for Carrying Out the Invention

Hereinbelow, the best mode of the present invention will be described in detail by comparing with comparative examples.

Example 1

In this example, a fine particulate silver powder was produced using the production method stated above and the properties of the obtained fine particulate silver powder were measured. And further, a silver paste was produced with the fine particulate silver powder and a test circuit was formed and the conductor resistance and sintering starting temperature were measured.

First, 63.3 g of silver nitrate was dissolved in 9.7 liters of pure water to prepare a silver nitrate aqueous solution, and 235 ml of 25 wt% concentration ammonia water was added thereto at once and agitated and a silver ammine complex aqueous solution was obtained.

This silver ammine complex aqueous solution was introduced into the first flow path a of 13 mm inside diameter shown in Figure 1 at a flow rate of 1,500 ml/sec and a reducing

agent was flowed from the second flow path b at a flow rate of 1,500 ml/ sec, and they were contacted at the joining point m at a temperature of 20°C and a fine particulate silver powder was reduced and deposited. A hydroquinone aqueous solution in which 21 g of hydroquinone was dissolved in 10 liters of pure water was used as the reducing agent on this occasion. Therefore, hydroquinone concentration at the time point when mixing was finished was about 1.04 g/l, and it was a very low concentration.

Filtration was performed with a Nutsche to separate the resulting fine particulate silver powder, which was washed with 100 ml of water and 50 ml of methanol and further dried at 70°C x 5 hours and a fine particulate silver powder was obtained. The photomicrograph of this obtained fine particulate silver powder by scanning electron microscope was shown in Figure 2.

The powder properties of the fine particulate silver powder obtained as above are shown in Table 1 along with the powder properties of the silver powders obtained in Example 2 and Comparative Examples. Therefore, measuring method and the like unidentified in the explanation described above are clarified here. The sintering starting temperature in Table 1 was measured by accurately weighing 0.5 g of a fine particulate silver powder with a balance and pressing this into a shape of a pellet at a pressure of 2 t/cm^2 for one minute and performing measurement with TMA/SS6000 which was a thermomechanical analysis equipment (TMA equipment) manufactured by SEIKO Instruments Corporation and in a

condition of air flow rate of 200 cc/min, temperature elevating rate of 2°C/min and retention time of 0 minute in the range of ordinary temperature to 900°C. The conductor resistance described in Table 1 was measured by using a 1mm width circuit obtained by producing a silver paste with each of the silver powders and drawing circuit on a ceramic substrate and sintering it to such a degree as allowing the measurement of resistance in the temperature range of 180 to 250°C. The composition of this silver paste was 85 wt% of a fine particulate silver powder, 0.75 wt% of ethyl cellulose, 14.25 wt% of terpineol. FIB analysis measured the dimension of deposited crystal particles and was used for measurement of crystallite diameter. The carbon content was to estimate the amount of impurities adhered to the powder particles of the silver powder and measured by mixing 0.5 g of a fine particulate silver powder, 1.5 g of tungsten powder and 0.3 g of tin powder and placing the mixture in a porcelain crucible and performing the measurement by burn up infrared absorption method using EMIA-320V manufactured by Horiba, Ltd.

Example 2

In this example, a fine particulate silver powder was produced using the production conditions different from those of Example 1 and the properties of the obtained fine particulate silver powder were measured. And further, a silver paste was produced with the fine particulate silver powder and a test circuit was formed and the conductor resistance and sintering starting temperature were measured.

First, 63.3 g of silver nitrate was dissolved in 3.1 liters of pure water to prepare a silver nitrate aqueous solution, and 235 ml of 25 wt% concentration ammonia water was added thereto at once and agitated and a silver ammine complex aqueous solution was obtained.

This silver ammine complex aqueous solution was introduced into the first flow path a of 13 mm inside diameter shown in Figure 1 at a flow rate of 1,500 ml/sec and a reducing agent was flowed from the second flow path b at a flow rate of 1,500 ml/sec, and they were contacted at the joining point m at a temperature of 20°C and a fine particulate silver powder was reduced and deposited. A hydroquinone aqueous solution in which 21 g of hydroquinone was dissolved in 3.4 liters of pure water was used as the reducing agent on this occasion. Therefore, hydroquinone concentration at the time point when mixing was finished was about 3.0 g/l, and it was a very low concentration.

The resulting fine particulate silver powder was filtered with a Nutsche as in Example 1, washed with 100 ml of water and 50 ml of methanol and further dried at 70°C x 5 hours and a fine particulate silver powder was obtained. The photomicrograph of this obtained fine particulate silver powder by scanning electron microscope was shown in Figure 3. The powder properties of the fine particulate silver powder obtained as above are shown in Table 1 along with the powder properties of the silver powders obtained in Example 1 and Comparative Examples.

Comparative Example 1

In this example, a fine particulate silver powder was produced using the production method shown below and the properties of the obtained fine particulate silver powder were measured. And further, a silver paste was produced with the fine particulate silver powder and a test circuit was formed and the conductor resistance and sintering starting temperature were measured.

First, 63.3 g of silver nitrate was dissolved in 1.0 liter of pure water to prepare a silver nitrate aqueous solution, and 235 ml of 25 wt% concentration ammonia water was added thereto at once and agitated and a silver ammine complex aqueous solution was obtained.

And this silver ammine complex solution was placed into a reaction tank and a hydroquinone aqueous solution in which 21 g of hydroquinone was dissolved in 1.3 liters of pure water was added thereto at once as the reducing agent and agitated while the liquid temperature was maintained at 20°C for allowing reaction and a fine particulate silver powder was reduced and deposited. The hydroquinone concentration at the time point when mixing was finished was about 8.23 g/l, and it was a high concentration.

The resulting fine particulate silver powder was filtered with a Nutsche as in Example 1, washed with 100 ml of water and 50 ml of methanol and further dried at 70°C x 5 hours and a fine particulate silver powder was obtained. The photomicrograph of this obtained fine particulate silver powder by scanning electron microscope was shown in Figure 4. The powder properties of the fine particulate silver powder

obtained as above are shown in Table 1 along with the powder

properties of the silver powders obtained in the

above-mentioned Examples and the second Comparative Example.

Comparative Example 2

In this example, a fine particulate silver powder was

produced using the production method shown below and the

properties of the obtained fine particulate silver powder

were measured. And further, a silver paste was produced with

the fine particulate silver powder and a test circuit was

formed and the conductor resistance and sintering starting

temperature were measured.

First, 63.3 g of silver nitrate was dissolved in 300

ml of pure water to prepare a silver nitrate aqueous solution,

and 235 ml of 25 wt% concentration ammonia water was added

thereto at once and agitated and a silver ammine complex aqueous

solution was obtained.

And this silver ammine complex solution was placed into

a reaction tank and a hydroquinone aqueous solution in which

3 g of gelatine was added to 200 ml of pure water and further

21 g of hydroquinone was dissolved in 700 ml of pure water

was added thereto at once as the reducing agent and agitated

while the liquid temperature was maintained at 20°C for

allowing reaction and a fine particulate silver powder was

reduced and deposited. The hydroquinone concentration at the

time point when mixing was finished was about 14.5 g/l, and

it was a high concentration.

The resulting fine particulate silver powder was filtered

with a Nutsche as in Example 1, washed with 100 ml of water

and 50 ml of methanol and further dried at 70°C x 5 hours and a fine particulate silver powder was obtained. The photomicrograph of this obtained fine particulate silver powder by scanning electron microscope was shown in Figure 5. The powder properties of the fine particulate silver powder obtained as above are shown in Table 1 along with the powder properties of the silver powders obtained in the above-mentioned Examples and the second Comparative Example.

Comparative Example 3

In this example, a fine particulate silver powder was produced using the production method shown below and the properties of the obtained fine particulate silver powder were measured. And further, a silver paste was produced with the fine particulate silver powder and a test circuit was formed and the conductor resistance and sintering starting temperature were measured.

First, 20g of polyvinylpyrrolidone was dissolved in 260 ml of pure water and further 50 g of silver nitrate was dissolved to prepare a silver nitrate aqueous solution, and 25 g of nitric acid was added thereto at once and agitated and a nitric acid solution containing silver was obtained. The ascorbic acid concentration at the time point when mixing was finished was about 36.0 g/l.

In the meantime, 35.8 g of ascorbic acid was added and dissolved to 500 ml of pure water as a reducing agent to prepare a reducing agent solution.

And this nitric acid solution containing silver was placed into a reaction tank and the above-mentioned reducing

agent solution was added thereto at once and agitated while the liquid temperature was maintained at 25°C for allowing reaction and a fine particulate silver powder was reduced and deposited.

The resulting fine particulate silver powder was filtered with a Nutsche as in Example 1, washed with 100 ml of water and 50 ml of methanol and further dried at 70°C x 5 hours and a fine particulate silver powder was obtained. The powder properties of the fine particulate silver powder obtained as above are shown in Table 1 along with the powder properties of the silver powders obtained in the above-mentioned Examples and Comparative Examples.

<Comparison and reviewing of Examples and Comparative Examples>

The above-mentioned Examples and Comparative Examples are compared referring to Table 1. In addition, it is supposed that the particle diameter of the primary particles of a powder particle can be understood distinctly if the scanning electron microscope photographs shown in Figure 2 to Figure 5 are referred to.

[Table 1]

Sample	Powder Properties							Properties of Sintered Conductor	
	SSA m²/g	Tap Bulk Density g/cm³	D$_{50}$	D$_{LA}$	D$_{50}$/D$_{LA}$	Crystallite Diameter nm	Carbon Content %	Conductor Resistance μΩ-cm	Sintering Starting Temperature°C
			μm						
Example 1	2.54	4.2	0.31	0.30	1.03	7	0.28	4.6	160
Example 2	1.68	4.7	0.55	0.49	1.12	7	0.32	5.9	190
Comparative Example 1	1.18	4.3	1.78	1.02	1.75	9	0.88	Not Measurable	250
Comparative Example 2	0.55	4.0	3.90	2.20	1.77	8	0.89	Not Measurable	250
Comparative	0.62	4.0	3.03	1.20	2.53	38	0.30	Not	350

Example 3								Measurable	

As is apparent from this Table 1, it will be appreciated that the fine particulate silver powders obtained in the above Examples are extremely fine and highly dispersible as compared with the silver powders produced with a conventional production method by comparing each of the powder property values and they are fine particulate powders never existed in the conventional silver powders. In addition, as for the sintered conductor properties, the film density in which the circuit is formed by using a fine particulate silver powder according to the present invention is high, and the electric resistance is reduced. In the case of each of the Comparative Examples, it can be seen that the conductor resistance is too high to be measured.

Industrial Applicability

The fine particulate silver powder according to the present invention is composed of fine powder particles as could never be supposed in the conventional silver powder, and the aggregation degree of the powder particle is low, and shows very excellent dispersibility as compared with the conventional silver powders. In addition, by adopting a production method of a fine particulate silver powder according to the present invention, the residual organic matter in the obtained fine particulate silver powder has been decreased, which effects along with the high film density

due to fine particulate silver powder and, as a result, enables
to reduce the electric resistance of the obtained conductor.